Diamonds for Use in Abrasive Tools

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***Abstract*— The development of technological processes for obtaining diamond powder is of industrial interest as a basic material in products for roughing and finishing surfaces, like in the polishing of ornamental rocks. This work investigates the influence of iron, as a doping agent, in association with the graphite to diamond transformation, which occurs during high pressure and high temperature synthesis in the presence of Ni-Mn as a catalyst-solvent metallic alloy. Diamond synthesis was carried out at 4.3 GPa of pressure and 1300°C using a reactive mixture with 1:1 ratio of graphite and Ni-Mn alloy powders doped with up to 5% of Fe. The results indicated that that diamonds produced in the presence of Fe are more friable with increasing broken percentage of crystals. The higher growth speed is apparently the responsible for the defects that reduces the crystal strength. The results of the bond retention test demonstrate clearly that the textured diamond particles are more likely to shear rather than to be pulled out from the metal matrix.**

***Index Terms*—Diamond synthesis, high pressure, crystal growth**

I. Introduction

Diamond diamond powders are mainly used in abrasive applications, in which diamond particles are bonded on a variety of tools for drilling, sawing, grinding, cutting and slicing applications[1]. The production of diamond tools requires that diamond particles be firmly retained in the bonding material in which they are embedded. The bonding between diamond particles and binder material (matrix) can be mechanical or chemical. The chemical bond is a result of diamond reaction with binder material at diamond-binder interface. Thus, the surface state of the crystals is very important. The greater the surfaces texture of the crystal, the better the capacity of its attachment to the matrix. Consequently, when bonded on a tool, the surface textured diamond particles are thought to provide increased bond retention compared to regular diamond particles [1]. Most industrially produced diamonds have smooth surface. To improve adhesion of the crystals in the matrix various etching methods have been developed [2-5]. However, this leads to increased production costs and not always these methods are effective. Additionally, these techniques do not provide a good control over the etching process and lead to changes in surface chemistry of diamond coupled with contamination of diamond surface. It is well established that quality of the crystals can be manipulated by controlling the diamond synthesis parameters. Modifying the rate of crystal growth changes in both crystal habit and the surface conditions are observed [6-8]. In this context, the objective of this paper is to present the researches results developed by the authors aimed to obtain diamonds with characteristics required for use in diamond tools.

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II. Experimental Procedure

The following materials were used as precursors for the synthesis of diamond powder: graphite with particle size of 400-600 μm, supplied by Unimetal; an alloy with 40 wt.% Ni and 60 wt.% Mn; supplied by the firm Vetec, Fe powder with particle size of 50-100 μm supplied by Aldrich. The powders were mixed in a fixed proportion of graphite to catalyst/solvent of 1:1. The Fe powder was incorporated into the reactive mixture in amounts up to 5 wt.%. After mixing the powders for one hour, the resulting reactive mixtures were pre-compacted in a cylindrical mold with 30mm in diameter at 200 MPa of pressure. The pre-compacted cylindrical samples were transferred to a deformable calcite capsule, forming a reactive cell, which was then mounted inside a toroidal anvil type of high pressure device (HPD) installed in a 2500ton hydraulic press. The HPHT synthesis was carried out at 4.7 GPa and 1300°C, as described elsewhere [3]. In short, initially the pressure was calibrated at room temperature using Bi sensors, with I to II transition at 2.55 GPa and PbSe at 4.3 GPa. It was expected a pressure variation inside the HPD chamber of the order of 5%. Temperature was calibrated afterwards using a type K chromel-alumel thermocouple diametrically inserted in the centre of the reactive cell. A correlation between the applied electrical current and the temperature was then performed. The effect of the pressure in the thermocouple electromotive force, emf, was not taken into consideration in the construction of the calibration curves.

The HPHT synthesis time was 10 mm after which pressure was released and the system allowed cooling to room temperature before opening the HPD. Agglomerates consisting of non-transformed graphite, metallic alloy, calcite and diamond crystals, was obtained. The diamond crystals were separated from the agglomerate by means of a purification process [9]. The pure crystals were then weighed and selected for their granulometry using standard sieves. The crystal mechanical strength was determined by

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friability tests in Ukrainian Frietester 1 equipment. For each friar test two carats (about 0.4 g) of diamonds were compressed until rupture. Morphology and structural properties of the synthesized crystals were characterized by scaning electron microscopy (SEM).

III. Results and Discussion

Figure 1 shows the results corresponding to the diamond synthesis productivity, Y, measured by the weight of crystal produced as a function of the Fe content.

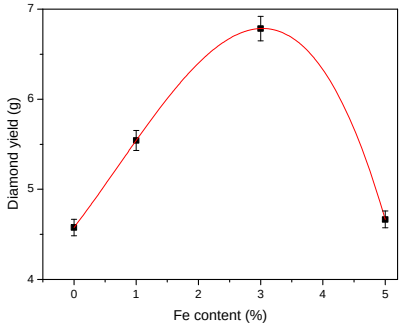


Figure 1 – Variation of diamond yield (Y) with Fe content.

In this figure, it should be noticed that lower yields are associated with $CaCO_3$ additions from 5.0 wt.%, which was related to the lowest productivity found in this work. The results in Fig. 1 indicate that a Fe addition up to 3.0 wt% causes an increase in the diamond yield. This can be attributed to a balance between the effect of Fe additions on the nucleation and growth of diamond crystals. Certainly, the interaction between the Fe with carbon atoms plays a role in this mechanism, which is, however, not well understood. It is suggested that the presence of the Fe interferes in the reactive mixture by decreasing the activation energy of the graphite to diamond transformation. Another possible reason could be the elevation in the catalyst effect of the Mn-Ni alloy. In any case, the Fe up to 3.0wt% acts to increase the synthesis yield.

Figure 2 shows the correlation between the diamond productivity and the different granulometric groups of crystal size for the distinct Fe contents. In this figure, one should pay attention to the fact that, independently of the Fe content, most diamond crystals were synthesized within the interval from 150 to 300 µm, with a maximum productivity corresponding to granulometric group 5, with 250/212 µm. It should be noted that this range of particle size is more used in the production of grinding tools, such as grinding wheels and dressers [1].

Figure 3 depicts SEM images of the diamond crystals obtained in the interval from 250 to 300 µm, corresponding to peaks in weight for the different doping content of Fe. Fig. 3a shows that the diamond synthesized in the Ni-Mn-C system (without Fe additions) are predominantly composed by the {100} and {111} faces and the surfaces are all flat and smooth.

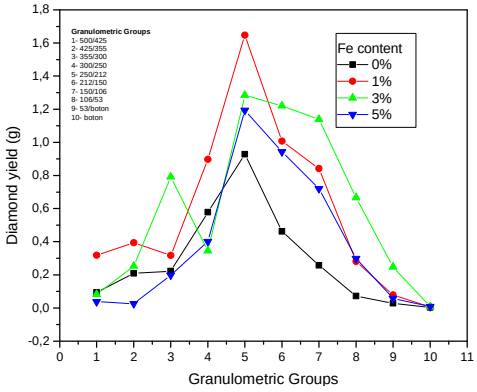
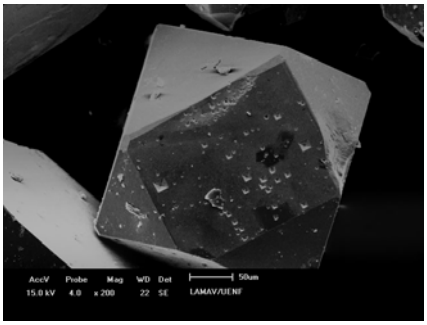
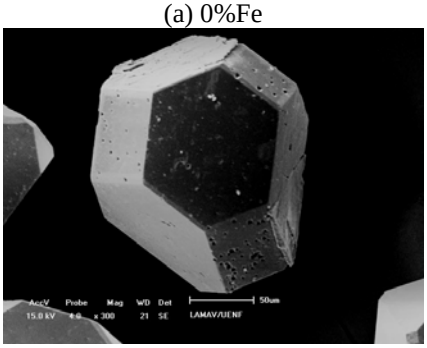


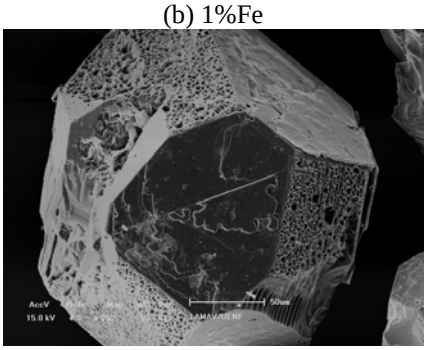
Figure 2 - Correlation between the diamond productivity and the granulometry of synthesized crystals for the different Fe contents.



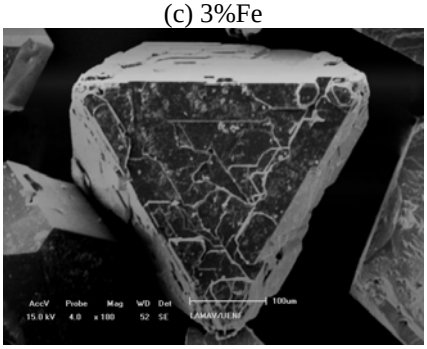
(a) 0%Fe



(b) 1%Fe



(c) 3%Fe



(d) 5%Fe

Figure 3 – SEM images of the diamond crystals as a function of Fe content.

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However, with the introduction of iron in the growth medium, the crystals begin to show significant changes in the surface. The surfaces of the diamond synthesized with Fe additive are rough and some growth layers are observed on the all faces, Fig. 3c and Fig. 3d. This type of surface might be help to improve the characteristics of diamond-coated tools [10]. The defects noted in surface result in the improvement of the adhesive ability of the crystals.

Friability is an important property of the crystals used in abrasive diamond tools. To determine the friability, it was chosen particle size with higher productivity both without addition and with the addition of Fe. Table I shows the percentage of broken diamond crystals, breaking ratio as a function of the Fe content. For the friar test, non-doped crystals with 150 to 212 µm and Fe doped crystals with 250 to 300 µm were selected. In Table I it is important to notice that the breaking ratio increases with doped Fe content. This reveals the brittle characteristics of the diamond crystals, which tends to be more severe with the incorporation of Fe up to 3wt% and is directly associated with the increase in surface and internal defects.

TABLE I
DIAMOND FRIABILITY RESULTS

% Fe	300/250	250/212	Breaking (%)	FI
0	0,202	0,198	49,5	0,0047
1	0,024	0376	94,0	0,0053
3	0,007	0,393	98,2	0,0054
5	0,130	0,270	67,5	0,0054

The bond retention of surfaces diamond particles obtained in present work compared with commercial diamond was assessed by tests of abrasion of the cutting discs made from these diamonds. After reaching 50% of tool life, the cutting surface was observed by confocal microscopy, Fig.4 and 5. Examination of the micrographs taken from cavities from which a diamond crystal was pulled out (white circles) reveals that while the pulled out of commercial diamond leaves a cavity with smooth and clean surface, the diamond particle (obtained in this work) leaves a cavity with a rough surface and debris is also present.

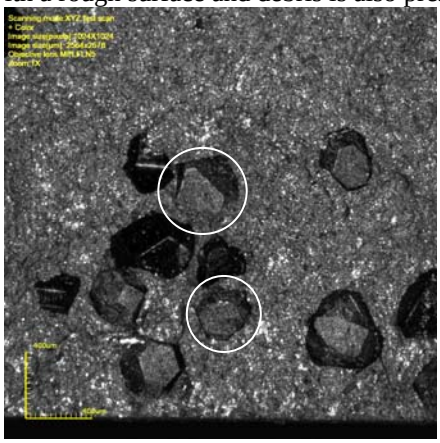

Figure 4 – Confocal micrograph of worn surface of a segment produced with commercial diamonds

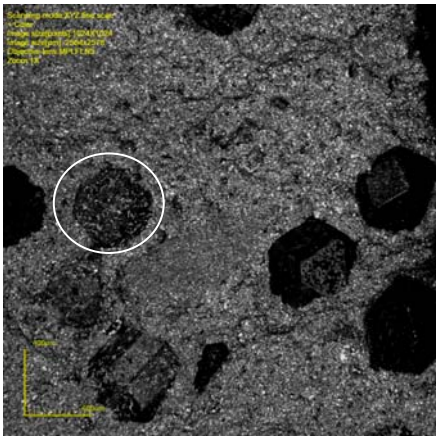

Figure 5 – Confocal micrograph of worn surface of a segment produced with diamonds obtained in this research

IV. CONCLUSION

The doping with Fe of a reactive mixture of graphite and Mn-Ni catalyst/solvent alloy sensibly affects the yield and quality of high pressure and high temperature synthesized diamond crystals.

The diamond yield significantly increases with doping content up to 3.0% of Fe, from 4.6g for non-doped reactive mixture to a maximum of 6.78g for doping with 3.0% Fe.

The granulometric distribution of diamonds is displaced towards higher crystal sizes with any amount of added Fe. A microstructural analysis showed crystals with more surface defects and lesser cubic morphology for doped reactive mixtures.

Friar tests indicate that diamonds produced in the presence of Fe are more friable with increasing broken percentage of crystals. The higher growth speed is apparently the responsible for the defects that reduces the crystal strength.

The results of the bond retention test demonstrate clearly that the textured diamond particles are more likely to shear rather than to be pulled out from the metal matrix.

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